A11102 129290

USE OF A MODIFIED ROSENHAIN FURNACE FOR THERMAL ANALYSIS

By H. Scott, and J. R. Freeman, jr.

CONTENTS

		Page
I.	Introduction	317
II.	Description of furnace	318
III.	Description of the elevating mechanism	318
IV.	Details of operation	321
V.	Summary	323

I. INTRODUCTION

In a paper presented before the Institute of Metals entitled "Some Appliances for Metallographic Research," Rosenhain [1] described a new type of furnace designed primarily for the thermal analysis of metals by the inverse-rate method and used by him with considerable success in the metallurgical department of the National Physical Laboratory. In his discussion of this type of furnace Rosenhain pointed out certain difficulties met with in its operation, such as uniformity of rate of heating or cooling being inadequate for the degree of accuracy desired. To overcome this difficulty, he suggested in place of motor propulsion a gravity drive controlled by a "hydraulic cylinder with a relief valve whose width of opening can be regulated to allow of any desired rate of motion." The authors in constructing a thermal-analysis furnace of Rosenhain's type have, therefore, followed his suggestion and also added certain features which increase somewhat the convenience and simplicity of its operation. Requests for information regarding this furnace and the highly satisfactory results obtained from its use justify, it is believed, describing its construction and operation in sufficient detail to make possible its duplication or improvement.

[1] Rosenhain, J., Inst. of Metals, 13, p. 160; 1915.

II. DESCRIPTION OF FURNACE

The details of the furnace construction are shown in Fig. 1, which is drawn to scale. The heating tube is of ¼-inch wall "alundum," heated at the upper end by 17 turns of 0.52-mm platinum wire, which is coated with alundum cement supplied for this purpose. The cement coating is essential when a temperature over 1000° C is required, as it prevents hot spots with the resulting burning out of the heater. This platinum-wire winding, unlike "nichrome," is entirely satisfactory for temperatures of at least 1000° C. It has been maintained at that temperature continuously for two months and shows no signs of deterioration. This temperature is maintained by a current of 5 amperes drawn from 30 volts potential; so its necessarily continuous operation is quite economical.

The furnace is heated at the top, as is Rosenhain's, to avoid convection currents, but the sample in its containing tube is introduced from the bottom, or cold end, thereby avoiding the disadvantages of his method, which consist of inconvenience in position of the sample and control apparatus and the heating of some portion of the sample tube at all times to the maximum temperature of the furnace. The latter disadvantage may prove serious in the event of slight inhomogeneities in the thermocouple wire.

III. DESCRIPTION OF THE ELEVATING MECHANISM

The details of the rate-control mechanism are shown in Fig. 2. The weights *K* (total weight, 15 pounds), operating over pulleys, lift the elevator *B*, and the weight *J* (weight, 2 pounds) lowers it. The rate of motion of the tube *C*, clamped on the elevator, is controlled by the flow of oil from one end of the cylinder *L* to the other through the needle valve *M*. The oil cylinder is kept open to the air and filled with a good grade of engine oil, care being taken that the oil is free from dirt and air bubbles, which might easily cause variations in the rate of motion of the plunger. The sample tube *C* is held and centered with three set screws in a sleeve *D*, which fits into a receptacle on the elevator, facilitating rapid changing of the sample. A guide rod *E* prevents rotation of the elevator and steadies its motion.

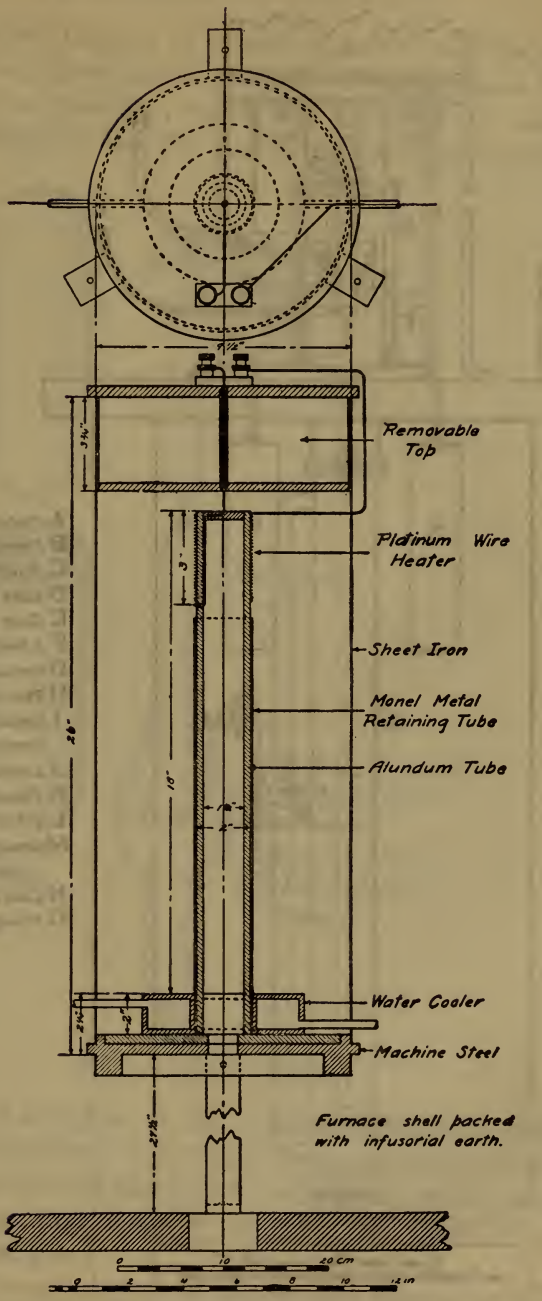

FIG. 1.—*Diagram of furnace*

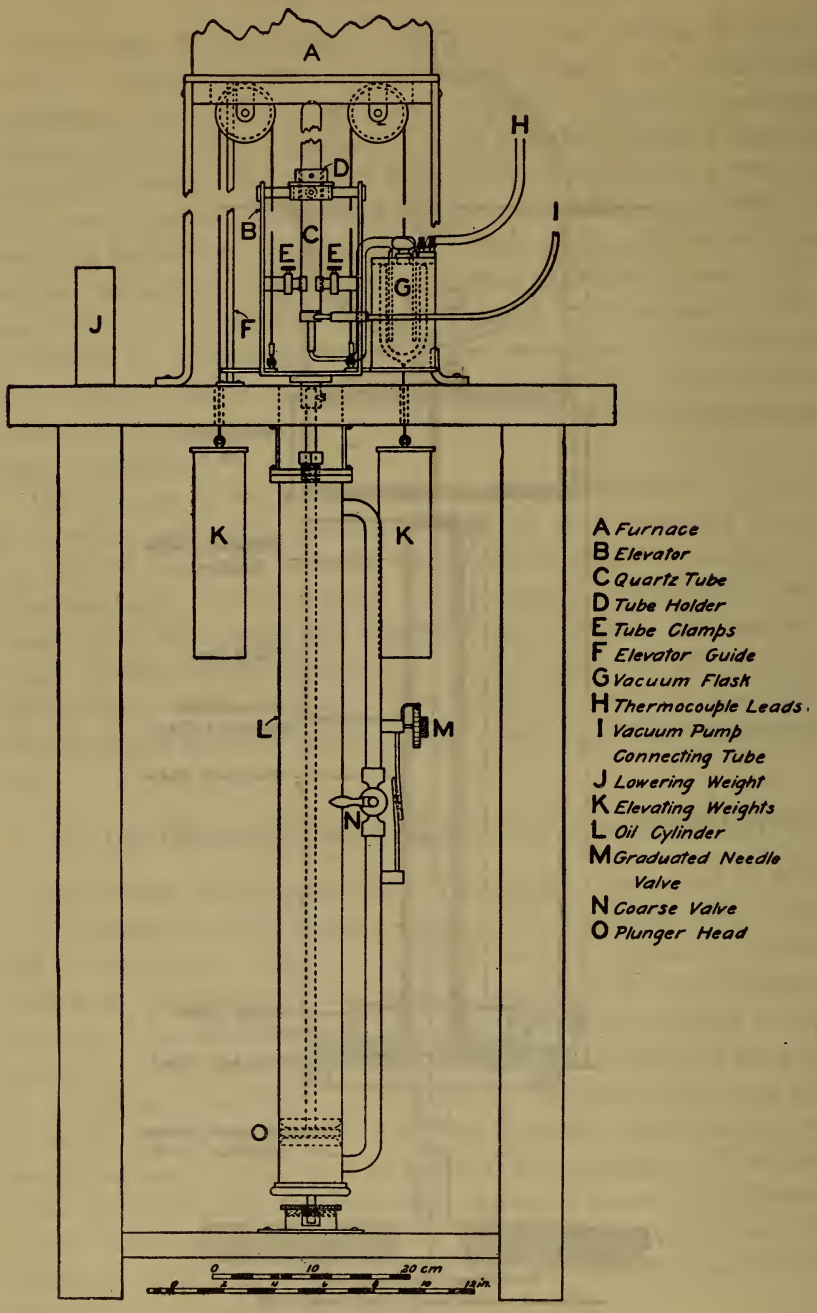

FIG. 2.—*Diagram of elevating mechanism*

IV. DETAILS OF OPERATION

The differential method of obtaining curves may be used with this furnace, but the experience of the authors has been that more valuable and satisfactory results are obtained by use of the inverse-rate method, and this has accordingly been used almost

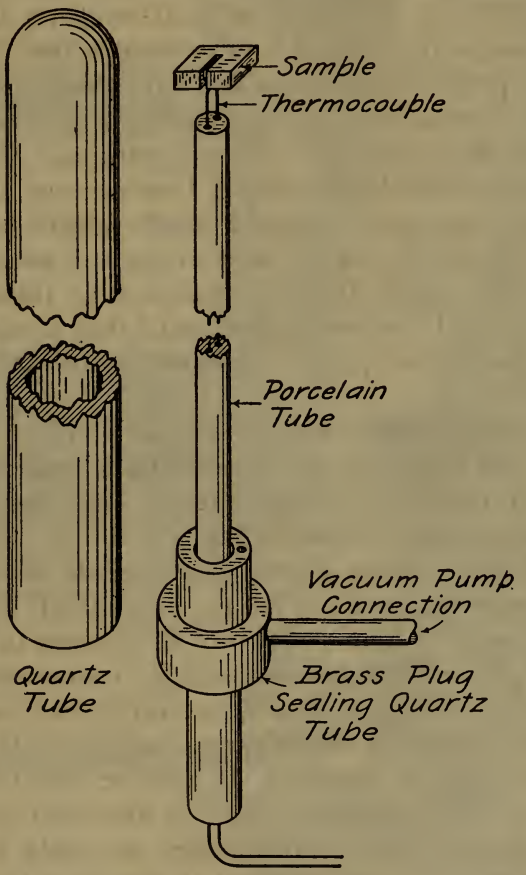

FIG. 3.—*Sample mounting*

exclusively. The adoption of the inverse-rate method limits the pyrometric requirements to a single thermocouple and potentiometer. This permits of the use of a somewhat novel method of mounting samples first used by Burgess and Crowe [2] in their researches on pure iron.

The aforementioned method of mounting is illustrated in Fig. 3. The operations involved consist simply of cutting a 0.5-mm slot in the sample with a small hack saw and riveting in this slot the

[2] Burgess and Crowe, The Critical Ranges A_2 and A_3 of Pure Iron, Bureau of Standards Scientific Paper No. 213.

flattened head at the hot junction of a platinum—90 platinum-10 rhodium thermocouple in the form of 0.5-mm-diameter wire. The mounted sample is sealed in the quartz tube and a vacuum maintained through the brass-plug connection.

This method of mounting has the advantages of good thermal contact between the sample and the thermocouple, use of small samples (usually $\frac{1}{8}$ by $\frac{9}{32}$ by $\frac{3}{8}$ inch) weighing about 1.7 g), with the consequent elimination of detectable thermal gradients, and ease of preparation of samples. Its chief disadvantage is the slight contamination of the thermocouple resulting from close contact with the sample at high temperatures. This source of error is easily avoided by using a homogeneous thermocouple and frequently removing the short length subject to contamination. A check can be had on the accuracy and sensitivity of the apparatus under actual operating conditions by taking curves on pure iron, the A_2 transformation of which has a maximum heat effect very constant at 768° C,[3] independent of rate of temperature change.

The temperature measurements are made with a dial potentiometer and the time interval recorded on a drum-type chronograph, which instruments have already been described.[4] The assembled apparatus is shown in Fig. 4.

A heating and cooling curve, characteristic of the furnace, taken on a transformationless (28 per cent nickel) steel over the temperature range of from 50 to 1000° C, is shown in Fig. 5, each curve being divided into two sections for convenience in reproduction. Curves of a steel showing several critical points of small intensity and taken with this apparatus are available in the work of one of the present authors.[5] It may be noted from Fig. 5 that the rate of temperature change is somewhat slower at the lower temperatures than at the higher, as would be expected, but that the change is not sufficient to obscure a transformation occurring anywhere between 100 and 1000° C. This change in rate is emphasized at the lower temperatures on the thermal curves of Fig. 5, due to the parabolic form of the relation between temperature and emf of the platinum couple, for the curves are plotted with time of unit emf change as abscissæ as a matter of

[3] Burgess and Crowe, loc. cit.

[4] Burgess and Crowe, loc. cit. Dr. P. D. Merica has substituted a pair of stop watches for the costly chronograph with good results, providing the time interval is greater than 15 seconds. Bull. A. I. M. & M. E. 151 p. 1021.

[5] Scott, H., Bureau of Standards Scientific Paper No. 335.

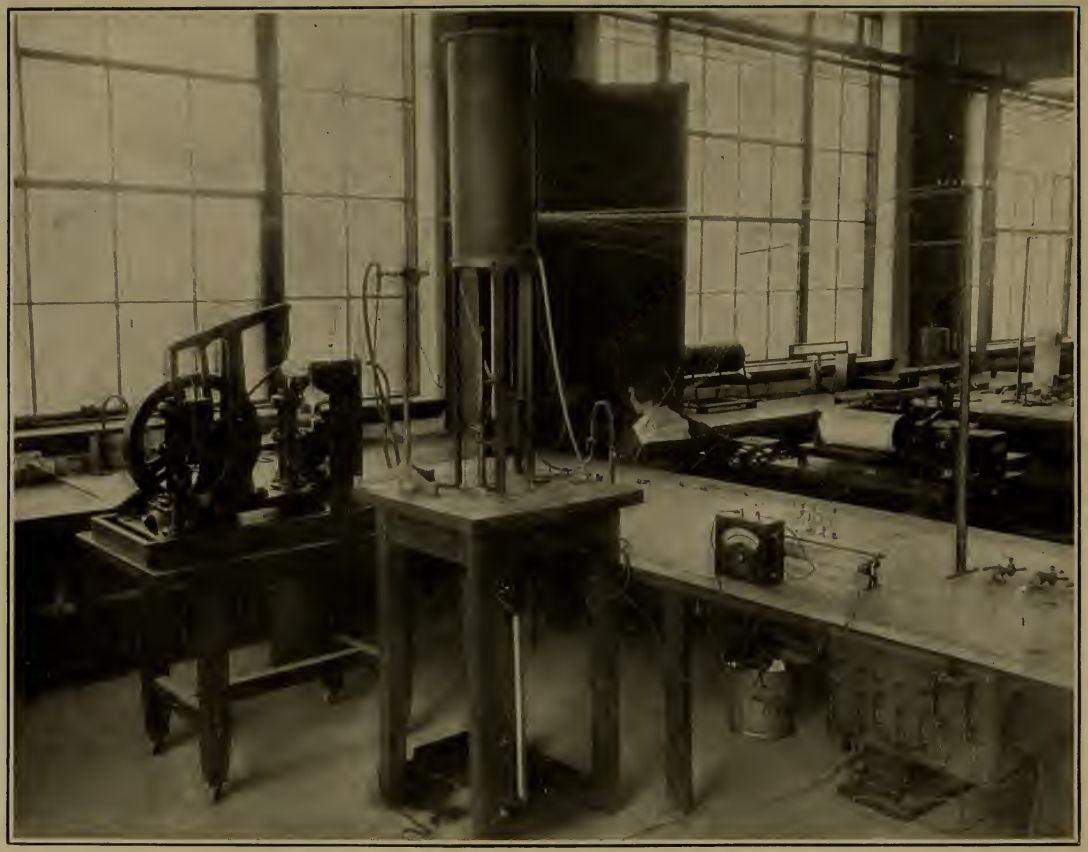

FIG. 4.—*Assembled furnace and auxiliary apparatus*

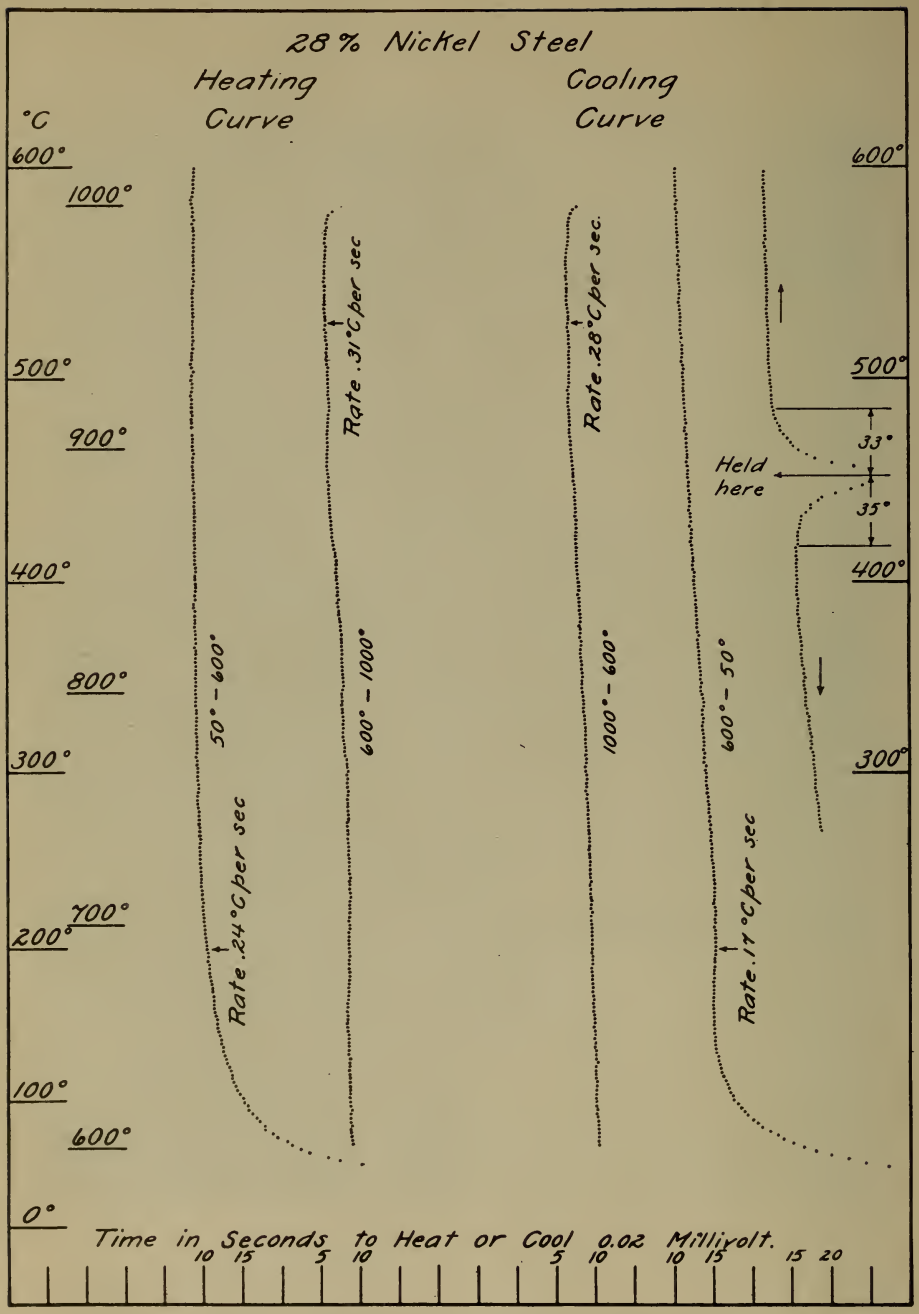

FIG. 5.—*Thermal curves of 28 per cent nickel steel*

convenience. The actual rate change can be reduced, and probably eliminated for a given rate; by using a metal cylinder or alundun tube tapered to increase the heat conduction at the lower temperatures.

It might be apprehended that the gravity drive would impart an extended acceleration to the elevator instead of a uniform velocity, but that the time required for the rate to become uniform is slight is shown by the short curves on the right-hand side of Fig. 4, obtained by bringing the sample to the constant temperature designated and then taking readings from the time of opening the valve. The time required for the rate to become normal for that valve setting is only 6½ minutes on heating and 4½ minutes on cooling, while the corresponding temperature interval is only 33 and 35° C. This is an extremely useful characteristic of the furnace, as it enables the separation of one transformation superimposed on the end of another by holding the sample at a temperature at which the first transformation will complete itself and then starting. It also facilitates the study of the effect of time of holding in the proximity of the transformation temperature on its position; that is, determining the limits of the transformation temperature at what amounts to zero rate of temperature change.

V. SUMMARY

A description is given of a thermal-analysis furnace constructed on the principle of Rosenhain's furnace, the chief departures from his design being: (1) Use of a gravity-drive rate control and (2) introduction of the sample through the bottom and cold end of the furnace. The advantages and faults of these modifications are mentioned.

The methods of operation developed and adopted for use with this furnace are described and discussed, the principal variation from the usual practice being the mounting of the sample in direct thermal contact with the thermocouple.

F. E. Mann contributed skill in the construction of this furnace and Miss H. G. Movius and H. A. Wadsworth assisted in its successful development.

WASHINGTON, D. C., May 28, 1919.